DETERMINATION OF CATION EXCHANGE CAPACITY AND ESTIMATION OF SMECTITE CONTENT IN ROCK SAMPLES USING METHYLENE BLUE ANALYSIS

Technical Implementing Procedure ID: OSTI-LBNL-TIP/GCT-1.0, Rev.0, Mod.0 *Effective: 10/04/04*

1. PURPOSE

This Technical Implementation Procedure (TIP) provides instructions to determine the cation exchange capacity (CEC) and estimate the smectite clay content of rock samples at Lawrence Berkeley National Laboratory (LBNL) for supporting the Office of Science & Technology and International (OSTI)-LBNL Project. The methylene blue method is best suited for analysis of smectite-bearing rocks.

2. SCOPE

This TIP applies to all OSTI-LBNL personnel (or contractor personnel following OSTI-LBNL procedures) involved in laboratory measurement of CEC and estimation of smectite content of rock samples using the methylene blue method for OSTI-LBNL activities subject to the U.S. Department of Energy (DOE) Office of Civilian Radioactive Waste Management (OCRWM) *Quality Assurance Requirements and Description* (QARD), DOE/RW-0333P. Prior to conducting work described in Section 3 of this procedure, personnel performing measurements require training in accordance with OSTI-LBNL Quality Implementing Procedure (QIP)-2.0, *Indoctrination and Training of Personnel*.

All technical activities, data collected using this procedure, and any equipment usage shall be in accordance with this TIP and in full compliance with OSTI-LBNL-QIP-12.0, *Control of Measuring and Test Equipment and Calibration Standards*. All documentation resulting from actions taken under this TIP shall be recorded in Scientific Notebooks and/or Equipment Log Books (which are controlled as a supplemental record) as described in OSTI-LBNL-QIP-SIII.0, *Scientific Notebooks*. While this procedure incorporates specific requirements unique to the laboratory studies, it is consistent with the requirements described in OSTI-LBNL-QIP-SII.0, *Documenting Sample Control*.

If this procedure cannot be implemented as written, OSTI-LBNL personnel shall notify the responsible Principal Investigator (PI) (or designee). If it is determined that a portion of the work cannot be accomplished as described in this TIP, or would produce undesirable results, that portion of the work shall be stopped and not resumed until this procedure is modified per OSTI-LBNL-QIP-5.0, *Preparing the Quality Assurance Plan and Quality/Technical Implementing Procedures*.

If the responsible PI (or designee) determines that a modification or a revision to the TIP would cause an unreasonable delay in proceeding with the task, then an expedited change to the procedure, including documentation of deviation from the approved procedure, can be made according to OSTI-LBNL-QIP-5.0. Such changes are subject to review, usually after the task has

proceeded, and thus work performed under TIPs with expedited changes is done at risk of future invalidation.

Scientific staff may use a controlled hard copy or an electronic "Information Only" copy of this procedure (available from the LBNL-ESD website); however, scientific staff are responsible for assuring that the correct revision of this procedure is used. When this procedure becomes obsolete or superseded, controlled copies must be destroyed or marked "superseded" in accordance with OSTI-LBNL-QIP-6.0, *Controlled Documents*, to ensure that this document is not used to perform work.

3. PROCEDURE

3.1 Principle

This procedure involves using the highly selective sorptivity of methylene blue on smectitic clay to determine the cation exchange capacity (CEC) and estimate the smectite content of bulk rock samples. The method, modified from the procedures listed in Stapel and Verhoef (1989) and Harvey et al. (2000), consists of (i) powdering the rock sample and weighing out an aliquot for analysis, (ii) adding the rock sample to a beaker containing H_2SO_4 and boiling the sample using a combined hot plate/magnetic stirrer, (iii) adding small increments of Methylene Blue (MeB) solution to the heated rock slurry, and after stirring, placing a drop of the rock suspension onto a piece of filter paper to determine whether there is a clear or blue ring around the slurry, and (iv) continue adding aliquots of the MeB solution to the rock suspension and testing the solution on the filter paper until the sorptive capacity of the powdered rock is exceeded, resulting in the formation of a blue ring around the rock residue . The amount of MeB solution that can be adsorbed by the rock mixture can be used to determine the CEC and estimate the amount of smectite clay present in the rock.

The responsible PI (or designee) decides the sample size and the incremental amount of MeB solution used for this measurement. Typically 0.5 g of powdered rock is used, and increments of 0.2-0.5 ml of MeB solution are pipetted into the rock slurry. These amounts shall be documented in the scientific notebook.

In brief, rock samples are:

A. placed in a drying oven at a temperature of 60±5°C for at least 1 hour to dry the samples prior to crushing;

B. subsequently crushed using a mortar and pestle or similar rock crusher to a grain size finer than 100 mesh;

C. a weighed aliquot of approximately 0.5 g rock powder is added to a 50 ml beaker;

D. 15 ml of 2N H_2SO_4 is added to the beaker, and the contents of the beaker are heated and stirred using a hot plate with a magnetic stirrer, so that the mixture is brought to a gentle boil for at least 2 minutes;

E. using a pipette, an aliquot of 0.2-0.5 ml of 0.01 N MeB solution (3.74 g/l of MeB in

water) is added to the mixture and allowed to mix with the rock slurry for 30 seconds;

F. a glass stirring rod is used to transfer a drop of the rock slurry onto a piece of filter paper. If the blue dye is retained by the powdered rock (and no blue ring develops in the wet spot around the rock), then the rock has not exceeded its capacity to adsorb MeB, and then step E is repeated. When the presence of a blue ring is noted, then wait another two minutes and repeat the drop test. If the blue ring persists, the test is now concluded, and the total amount of MeB added to the solution should be recorded. If the blue ring is no longer visible, repeat step E again.

All documentation resulting from actions taken under this TIP shall be recorded in the scientific notebook and/or Equipment Log Books.

3.2 Equipment

The list of equipment needed to conduct this procedure is shown below. Items equivalent to those listed below may be used provided they perform the same function with an acceptable level of performance as judged by the responsible PI (or designee). Any equivalent items used shall be documented in the scientific notebook in accordance with OSTI-LBNL-QIP-SIII.0.

- rock hammer
- mortar and pestle or similar rock crusher to powder rock samples
- high quality 100-mesh laboratory sieve in good condition
- calibrated top-loading electronic balance to measure out aliquots of ~1g of powdered rock samples. The balance should be tared using clean weighing paper prior to adding the powdered rock sample. The balance shall be calibrated in accordance to OSTI-LBNL-TIP/HT-5.0, *Balance Calibration*. The balance shall have a precision of 0.01 g or better. A higher precision balance (0.001 g or better) will be used for verification of pipette volumes.
- convection oven capable of maintaining the temperature at 60 ±5°C; oven temperature is measured using calibrated thermometers
- sample holders with tight-fitting lids (e.g., glass bottles) for storing excess powdered rock samples
- aluminum trays for drying samples in oven
- thermometer to monitor oven temperature (see Section 3.3.1 for information regarding calibration)
- hot plate with magnetic stirrer
- glass stirring rod for applying rock slurry samples to filter paper
- volumetric flask for preparing MeB solution (see Section 3.3.1 for information

regarding verification of accuracy)

- pipettes or pipettors for adding aliquots of acid and MeB solution (see Section 3.3.1 for information regarding verification of accuracy)
- 50 ml glass beaker
- weighing paper
- filter paper (Whatman No. 40 or equivalent) for conducting blue spot test
- tape and indelible marking pen to label sample bottles
- pencil for labeling filter paper
- laboratory timer to monitor timing of each step

Good laboratory and scientific practices shall be used to protect against operator injury. Be familiar with the material safety data sheets (MSDS) for each chemical to become familiar with chemical hazards and proper handling procedures. To prevent burns, use tongs, potholders, or the equivalents when removing rock samples from the oven. Wear safety glasses during powdering of rock samples and when working with acid. Appropriate safety garb (gloves, smock or lab coat) shall also be worn when handling acid.

3.3 Preparatory Verification

3.3.1 Calibration of Measuring and Test Equipment

Measuring and test equipment (M&TE) usage and calibration shall be in full compliance with OSTI-LBNL-QIP-12.0. Balances must be calibrated in accordance to OSTI-LBNL-TIP/HT-5.0. The thermometer used to monitor the drying oven temperature shall be calibrated by an outside vendor procured from OCRWM approved contractors on the Qualified Suppliers List (QSL), as per OSTI-LBNL-QIP-4.0, *Procurement Document Control*. The thermometer shall have a range that at least covers 0-100°C, with an accuracy of ±2°C. Verify the accuracy of volumetric flasks, pipettes and pipettors by weighing water quantities delivered or contained using a calibrated balance. Do not use flasks, pipettes and pipettors that differ by more than 1% of their nominal value. All glassware used for this procedure should be cleaned and rinsed thoroughly with deionized water prior to use.

The chemicals (sulfuric acid and methylene blue) shall also be procured from OCRWM-approved contractors on the QSL, using reagent grade chemicals that meet American Chemical Society (ACS) specifications if available. Certificates of analyses for these chemicals shall be obtained from the vendor, if available, and entered into the scientific notebook. Prepare the starting MeB solution by weighing out the appropriate amount of MeB (3.74 g/l of MeB in water) and adding it to a volumetric flask with deionized water, filling the flask up to the calibrated volume after adding the MeB to a partially filled flask.

The calibration intervals (e.g., annually or biannually) for the equipment, which are dependent upon the characteristics and usage intensity of the equipment, are decided by the PI (or designee). The balance operation should be verified prior to each use using a known weight. Balance calibration should be conducted annually in accordance with OSTI-LBNL-TIP/HT-5.0. The thermometer calibration interval is every two years. Verification of the accuracy of volumetric flasks, pipettes, and pipettors should be conducted annually. Verify that their calibration is still valid before using the M&TE.

3.3.2 Environmental Conditions

This TIP is performed in the laboratory environment under normal laboratory conditions. Observe and correct factors, if applicable, that may be detrimental to good weighing and sample preparation, such as dust, vibrations, air drafts, and temperature fluctuations.

3.4 Control of Samples

It is imperative that sample identification and control be sufficient to trace a sample and its derivatives from its original field location to the point of analysis. For samples collected in the field, a unique identifier is assigned to the sample by the Sample Management Facility (SMF), in accordance with AP-SII.2Q, *Requesting, Transferring, and Returning Yucca Mountain Project Geologic Borehole Specimens*. Traceability to this number shall be maintained even if a local identification number is assigned to the sample by recording the field-assigned identifier and the locally assigned identification in the scientific notebook.

3.5 Implementing Procedure

A. Initial Sample Preparation

Samples collected from the field for MeB analysis are normally received in plastic sample bags with unique sample identifications (e.g., SMF sample ID, borehole ID, depth interval).

1. Fill in the applicable items (such as SMF sample ID, borehole ID and depth interval) on the Data Sheet (Attachment 1) or enter equivalent information in the scientific notebook. Include Attachment 1, if used, in the scientific notebook.

2. Open the sample bag and select a representative portion of the sample for drying and crushing. Use a rock hammer if necessary to break off small fragments. Before processing the sample, make sure that it is free of any residual drilling mud. If the sample appears to contain drilling mud, wash the sample thoroughly to remove any surface contamination.

3. Place the selected rock fragments on a clean, labeled aluminum tray and insert in a 60 ±5°C convention oven for at least 1 hour to dry samples prior to crushing.

For samples that are received dry, 1 hour is sufficient. Wet or saturated samples may require additional drying. A number of samples can be dried simultaneously.

4. Using a clean mortar and pestle (or similar rock crushing apparatus), crush rock fragments from a single rock sample into a fine powder (finer than 100 mesh). A clean, high quality laboratory 100-mesh laboratory sieve in good condition (sieve calibration is not needed) may be used to check that the rocks are crushed sufficiently. Store the powdered sample in a glass bottle (or similar container) labeled with a sample ID number that is linked to SMF sample ID as appropriate.

B. Methylene Blue Analysis

1. Pipette ~15 ml of 2 N H_2SO_4 into a 50 ml glass beaker.

2. Using a top-load balance, first tare balance with weighing paper, and then add about 0.5 g of rock powder onto the paper. Record the weight of the sample. Add the sample to the beaker with acid.

3. Place the beaker with the sample and acid onto a hot plate with a magnetic stirrer, and heat until the solution reaches a gentle boil. Boil gently for at least 2 minutes.

4. Pipette an increment (0.2 to 0.5 ml) of 0.01 N MeB solution into the solution. Larger incremental volumes of MeB are best suited for smectite-rich samples. Record the amount pipetted into solution, and allow it to mix for 30 seconds.

5. Dip a glass stirring rod into the rock slurry and daub the filter paper with the tip of the rod so that a small amount of the suspended rock sample is applied to the paper. Note whether the wet ring on the filter paper that forms around the dyed solid residue is clear or blue. If it is clear, repeat steps 4 and 5. Label each drop test on the filter paper using a pencil.

6. When the ring finally turns blue, this indicates that the CEC of the rock sample has been exceeded. To verify this, wait another 2 minutes, and then repeat step 5. If the blue ring persists, then the end point has been reached. If there is no longer a blue ring, repeat steps 4 and 5.

7. When the end point has been reached, record the total amount of MeB solution that was added to the rock slurry. Report the results in ml MeB solution per gram of rock, and as grams of MeB per 100 g of rock sample. A 0.01 N MeB solution contains 3.74 g MeB per liter.; thus the number of grams of MeB per 100 g of rock sample is equivalent to the number of ml solution/g rock times 0.374.

C. Calculation of Cation Exchange Capacity

The CEC of the sample can be directly computed from the results of the above test.

This value is typically reported as meq MeB/100 g rock sample. The solution used is 0.01 N MeB, so each ml of 0.01N MeB added per gram of rock is equivalent to a CEC increment of 1 meq/100 g of rock.

D. Estimation of Smectite Content of Rock Sample

The group of smectite clay minerals has a wide range in chemical compositions that result in a range in CEC values. Kahr and Madsen (1995) report values ranging from 0.62-1.20 meq/g, and Gunderson et al. (2000) give a range from 0.8-1.5 meq/g. For estimating the smectite content of rock samples, we have adopted a representative CEC value of 100 meq/100 g of smectite (e.g., Gunderson et al., 2000). Because of the high selectivity of MeB to smectite (Gunderson et al., 2000), we also assume that all of the adsorption of MeB in the rock sample is due to the presence of smectite. Thus the smectite content of the rock (in percent) would be equivalent to the CEC value.

3.6 Potential Sources of Error and Uncertainty

One important source of uncertainty lies in selecting representative sample fragments for producing the powdered rock sample. If the original sample is heterogeneous, then care needs to be taken to select sample fragments that are representative of the bulk sample. An alternative approach is to sample different portions of a bulk sample (for example, the matrix and vein minerals as separate samples), and to determine if they have different smectite clay contents.

Another potential source of error and uncertainty in this method is that there is a range of values for the CEC of smectite, resulting from the chemical variability of this group of clay minerals (Meier and Nüesch, 1999). The estimation of smectite clay content using this method assumes a CEC value of 100 meq/100 g of smectite (the value used by Gunderson et al., 2000).

This method also assumes that smectite is the only mineral exchanging with the methylene blue dye. Because MeB selectively adsorbs to smectite, and has only minor sensitivity to zeolites, chlorite, and kaolinite (Gunderson et al., 2000), this assumption should not lead to large errors. Because of the aforementioned uncertainties, the CEC value for the rock sample should be considered to be the reliable result of this test, and the calculated smectite abundance should be considered to be an estimated value only. Independent semiquantitative (non-Q) verification of the abundance of smectite minerals can be obtained using X-ray diffraction techniques for clay minerals.

Another potential source of error to this test is the possibility of contamination of core and cuttings samples from residual drilling mud. Many drilling fluids contain bentonite, a form of smectite, and if drill samples have not been properly cleaned prior to analysis, contamination from the drilling bentonite could result in a false indication of the presence of smectite in the rock samples.

If a problem occurs which could pose a potential source of error or uncertainty for the results, then the staff member shall document it in the scientific notebook and contact

the PI about the problem. Samples that have characteristics that could be considered to be nonconforming to this procedure should be identified as nonconforming and a nonconformance report should be completed as per OSTI-LBNL-QIP-15.0, *Nonconformances*.

3.7 M&TE Storage and Handling

M&TE shall not be handled in a manner that adversely affects its current or future performance. M&TE shall be used in laboratory environments, and stored at room temperature.

3.8 M&TE Usage

Scientific staff shall document each usage of the M&TE in the scientific notebook (containing the same information as described in OSTI-LBNL-QIP-12.0) or the M&TE Standard Usage Log as described in OSTI-LBNL-QIP-12.0, and include the form in the scientific notebook. Calibration verification of the M&TE (Section 3.3.1) shall be conducted according to the required frequency.

3.9 Controls for Out-of-Calibration Conditions

If any out-of-calibration conditions (as described in OSTI-LBNL-QIP-12.0) are determined to exist for the M&TE item (e.g., instrument produces results known to be in error), the instrument shall have an out-of-service tag applied indicating that it is not to be used and, when possible, the instrument shall be moved to a segregated "out-of-service" area.

The above conditions shall be documented by using the M&TE Out of Calibration Report (OCR) in accordance with the instructions provided in OSTI-LBNL-QIP-12.0. If it is determined that the data are impacted, a Nonconformance Report (NCR) shall be initiated in accordance with OSTI-LBNL-QIP-15.0, *Nonconformances*.

4. RECORDS

The records listed in Section in 4.1 shall be collected and submitted the Records Coordinator for submittal to the OCRWM, in accordance with OSTI-LBNL-QIP-17.0, *Records Management,* as individual records or included in a records package.

4.1 QA Records

Records generated as a result of this TIP are entries in:

- Scientific notebooks or attachments to such notebooks

- Equipment Logbooks (including M&TE Standard Usage Log, if applicable)
- M&TE Out of Calibration Report, if applicable

4.2 Non-QA Long Term Records

None.

4.3 Non-QA Short-Term Records (three years or less retention)

None.

5. RESPONSIBILITIES

5.1 The **Principal Investigator (PI)** is responsible for assuring full compliance with this procedure and providing training thereof. The PI is responsible for overseeing and coordinating the preparation, review, distribution, revision, and recommending rescission of the TIP.

5.2 **Scientific Staff** are responsible for following this procedure and turning over related documentation to the Records Coordinator for submittal to the OCRWM, in accordance with OSTI-LBNL-QIP-17.0. Related data shall be turned over to Technical Data Coordinator in accordance with OSTI-LBNL-QIP-SIII.3, *Submittal and Incorporation of Data to the Technical Data Management System*, for entry into the Technical Database Management System (TDMS).

6. ACRONYMS AND DEFINITIONS

6.1 Acronyms

ACS	American Chemical Society
CEC	Cation Exchange Capacity
DOE	Department of Energy
ESD	Earth Sciences Division
GCT	Geochemical Testing
HT	Hydrogeological Testing
LBNL	Lawrence Berkeley National Laboratory
MSDS	Material Data Safety Sheet
M&TE	Measuring and Test Equipment

MeB	Methylene Blue
NCR	Nonconformance Report
OCR	Out of Calibration Report
OCRWM	Office of Civilian Radioactive Waste Management
OSTI	Office of Science & Technology and International
PI	Principal Investigator
QA	Quality Assurance
QARD	Quality Assurance Requirements and Description
QIP	Quality Implementing Procedure
QSL	Qualified Suppliers List
SMF	Sample Management Facility
TDMS	Technical Data Management System
TIP	Technical Implementing Procedure

6.2 Definitions

Cation Exchange Capacity (CEC): The ability of a rock or mineral to exchange cations with a fluid. This value is typically reported in units of milliequivalents of cation per 100 g of sample material.

Smectite: Dioctahedral (montmorillonite) and trioctahedral (saponite) clay minerals that possess swelling properties and high cation-exchange capacities.

Methylene Blue: The organic dye $C_{16}H_{18}N_3SCl \cdot 3H_2O$ (MW = 373.9) used for determination of cation exchange capacity of rocks and minerals.

Methylene Blue Adsorption: The amount of methylene blue that is adsorbed by a rock or mineral (expressed as g MeB per 100 g rock).

Rock Samples: Geologic materials intended for laboratory studies or analyses that constitute part of the OSTI-LBNL research program. Rock samples may include, but are not limited to, surface samples, drill cores, drilling cuttings, and rocks from underground excavations. Derivatives of rock samples collected from the field are also included in this definition.

Technical Implementing Procedure: Each TIP describes OSTI-LBNL technical tasks that (1) are repetitive, (2) are standardized, and (3) can return different results if

deviation from the sequence of steps occur.

7. REFERENCES

DOE/RW-0333P, *Quality Assurance Requirements and Description*

AP-SII.2Q, *Requesting, Transferring, and Returning Yucca Mountain Project Geologic Borehole Specimens*

Gunderson, R., Cumming, W., Astra, D., and Harvey, C. 2000. Analysis of Smectite Clays in Geothermal Drill Cuttings by the Methylene Blue Method: For Well Site Geothermometry and Resistivity Sounding Correlation, Proceedings of the World Geothermal Congress 2000, Kyushu – Tohoku, Japan, May 28–June 10, 2000, pp. 1175-1181.

Harvey, C.C., Gunderson, R., and Cumming, W. 2000. Methylene Blue Adsorption: A Real Time Rig Geologist Tool for Estimating Geothermal Reservoir Temperatures and Forecasting Drillhole Stability Problems, Proceedings of the 22nd New Zealand Geothermal Workshop, University of Auckland Geothermal Institute, Auckland, New Zealand, pp. 151-155.

Kahr, G., and Madsen, F.T. 1995. Determination of the cation exchange capacity and the surface area of bentonite, illite and kaolinite by methylene blue adsorption. Applied Clay Science, v. 9, pp. 327-336.

Meier, L.P, and Nüesch, R. 1999. The Lower Cation Exchange Capacity Limit of Montmorillonite, Journal of Colloid and Interface Science, v. 217, pp. 77-85.

OSTI-LBNL-QIP-2.0, *Indoctrination and Training of Personnel*

OSTI-LBNL-QIP-4.0, *Procurement Document Control*

OSTI-LBNL-QIP-5.0, *Preparing the Quality Assurance Plan and Quality/Technical Implementing Procedures*

OSTI-LBNL-QIP-6.0, *Controlled Documents*

OSTI-LBNL-QIP-12.0, *Control of Measuring and Test Equipment and Calibration Standards*

OSTI-LBNL-QIP-15.0, *Nonconformances*

OSTI-LBNL-QIP-17.0, *Records Management*

OSTI-LBNL-QIP-SII.0, *Documenting Sample Control*

OSTI-LBNL-QIP-SIII.0, *Scientific Notebooks*

OSTI-LBNL-QIP-SIII.3, *Submittal and Incorporation of Data to the Technical Data Management System*

OSTI-LBNL-TIP/HT-4.0, *Balance Calibration*

Stapel, E.E, and Verhoef, P.N.W. 1989. The Use of the Methylene Blue Adsorption Test in Assessing the Quality of Basaltic Tuff Rock Aggregate, Engineering Geology, v. 26, pp. 233-246.

8. ATTACHMENTS

Attachment 1 - Data Sheet for “Methylene Blue Analysis of Rock Samples”.

9. REVISION HISTORY

10/04/04 Revision 0, Modification 0

Initial Issue.

10. APPROVAL

(Signature on File) _____ _____

Preparer/PI: Patrick Dobson Date

(Signature on File) _____ _____

Technical Reviewer: Timothy J. Kneafsey Date

(Signature on File) _____ _____

Technical Reviewer: Guoxiang Zhang Date

(Signature on File) _____ _____

QA Reviewer: Vivi Fissekidou Date

(Signature on File) _____ _____

Project Manager: Gudmundur S. Bodvarsson Date

Methylene Blue Analysis of Rock Samples					
SMF sample ID:					
Borehole ID:					
Depth interval (m):					
Local sample ID:					
Date and time sample placed into the oven:					
Date and time sample taken out from oven:					
Date and time sample crushed to powder:					
Measurement of Sample Weight		Date	Time	weight (g)	Item
Weight of powdered rock (should be around 0.5 g)					A
Measurement of Acid		Date	Time	amount (ml)	
Volume of 2N H_2SO_4 added to beaker (should be around 15 ml)					B
Measurement of MeB	Date	Time	amount (ml)	Blue ring? (Y/N)	
Volume of first aliquot of MeB added					C
Volume of second aliquot of MeB added					D
Volume of third aliquot of MeB added					E
Volume of fourth aliquot of MeB added					F
Volume of fifth aliquot of MeB added					G
Volume of sixth aliquot of MeB added					H
Volume of seventh aliquot of MeB added					I
Volume of eighth aliquot of MeB added					J
Volume of ninth aliquot of MeB added					K
Volume of tenth aliquot of MeB added					L
Volume of eleventh aliquot of MeB added					M
Volume of twelfth aliquot of MeB added					N
Volume of thirteenth aliquot of MeB added					O
Volume of fourteenth aliquot of MeB added					P
Volume of fifteenth aliquot of MeB added					Q
Volume of sixteenth aliquot of MeB added					R
Volume of seventeenth aliquot of MeB added					S
Volume of eighteenth aliquot of MeB added					T
Volume of nineteenth aliquot of MeB added					U
Volume of twentieth aliquot of MeB added					V
Total volume of MeB added					W
Comments/Notes					